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FUNDAMENTAL INVESTIGATION OF MOLYBDENUM DISULFIDE
AS A SOLID LUBRICANT

Quarterly Progress Report No. 3

Contract NOw 64-0545-f

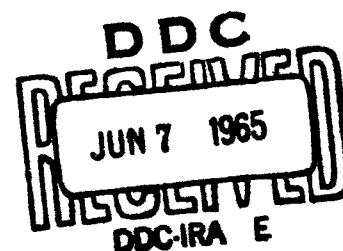
J. C. Tyler
P. M. Ku

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Report No. RS-451

FUNDAMENTAL INVESTIGATION OF MOLYBDENUM DISULFIDE
AS A SOLID LUBRICANT

Quarterly Progress Report No. 3

Contract NOw 64-0545-f

J. C. Tyler
P. M. Ku

to

Bureau of Naval Weapons
Department of the Navy
Washington, D. C. 20360

May 22, 1965

APPROVED:



P. M. Ku, Director
Department of Aerospace
Propulsion Research

FOREWORD

This report was prepared at Southwest Research Institute under Contract NOw-64-0545-f. The work was administered by the materials Division, Bureau of Naval Weapons, Department of the Navy, Washington, D. C. 20360, under the direction of Mr. T. McGee serving as project engineer.

This report covers the work performed during the period from January 23 through April 22, 1965.

Grateful acknowledgment is due Mr. M. J. Devine of Aeronautical Materials Laboratory, Naval Air Engineering Center, Philadelphia, Pennsylvania 19112, for his keen interest in this program and for his many helpful comments and suggestions as well as some supporting laboratory work.

Acknowledgment is also due Mr. K. B. Wood, Jr., of Climax Molybdenum Company, New York, New York 10020, through whose interest and courtesy, Climax Molybdenum Company will furnish to the program, without cost, a supply of molybdenum disulfide powder of known history and properties, to be used in the formal portion of the investigation.

Dr. R. A. Burton of the Institute staff and Professors F. F. Ling and W. E. Campbell of Rensselaer Polytechnic Institute participated in the program as consultants.

ABSTRACT

This report describes the third three months of work on an investigation of molybdenum disulfide (MoS_2) as a solid lubricant. The objective, scope, and program in general are briefly reviewed. A reference powder of documented composition and particle size distribution has been used for making a limited number of preliminary compression tests. The relation between specimen fracture stress, specimen specific gravity, and specimen length-to-diameter ratio has been investigated and is discussed. The variations of specimen specific gravity and hardness in the longitudinal direction have also been studied and the results are presented. A crack developed in the four-piece tapered sleeve of the compacting die during compact pressurization, and a new sleeve is in the fabrication stage. Fabrication and assembly of the test apparatus has been completed, and it is being instrumented and calibrated for subsequent work.

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I. INTRODUCTION

A. General

This report covers the third three months of effort on a fundamental investigation of molybdenum disulfide (MoS_2) as a solid lubricant, under Contract NOw 64-0545-f. At high temperatures, low temperatures, high vacuums, or under other extreme conditions of operation, the use of fluid-film lubrication is often infeasible. Under these circumstances the use of solid-film lubrication generally becomes mandatory. While progress of solid-film lubricant development has been slow, a basic understanding of the mechanism of solid-film lubrication is even more lacking. Therefore, this basic study of the mechanism of solid-film lubrication has been undertaken. The choice of MoS_2 in this investigation was prompted by its wide current use and future potential, as well as its seemingly complex behavior so that an understanding of its lubrication mechanism will, it is hoped, shed light on our knowledge of friction and wear phenomena in general.

B. Program and Approach

A research program of the type indicated must involve, of necessity, measurements of the physical and chemical properties of MoS_2 , studies of the physical and chemical interactions between MoS_2 and the metal substrates, and finally elucidation of the friction and wear mechanisms in the light of the measurements and studies made. An investigation of such comprehensive scope and challenging nature cannot feasibly, or even possibly, be accomplished at once. Therefore, a progressive approach has been adopted, in an effort to gain understanding in a step-by-step manner.

For the current contract period, it is expected that measurement of the basic properties of pressurized MoS_2 compacts, in a dry-air environment, will be completed, and selected runs at moderate vacuum (10^{-6} torr) will be made. The basic properties of primary interest are compressive, tensile, and shear strengths and cohesion, adhesion, and friction coefficients as well as specimen densities and hardnesses. The secondary tasks under consideration during this contract period are a design study of a method for experimental visualization of the sliding process and a critical study of available data on possible chemical interactions during the sliding process. In view of the large amount of work remaining

to be done, the experimental visualization of the sliding process will not be attempted during the current year. Also, it is anticipated that time will not permit an extensive study of the effects of chemical interactions. These aspects of the problem will be examined in light of data to be obtained from basic measurements of properties of MoS₂ compacts and will be considered in formulating a future program.

II. TEST SPECIMENS

A. Molybdenum Disulfide Powder

The importance of employing MoS₂ powder of documented processing history, composition, and particle size distribution for the basic measurements was emphasized in Progress Report No. 1. Accordingly, arrangements were made with Climax Molybdenum Company to furnish without cost a single batch of powder meeting the requirements of Military Specification MIL-M-7866, with complete documentation on all pertinent details, for use in this program. The material will be packaged in a number of sealed, one-pound cans, to be opened for use as needed. Care will be taken to insure uniform composition and particle size distribution among the cans. Most of the cans will be filled with high-purity nitrogen to insure storage stability. However, a small number of cans will also be packed in high vacuum, to serve as controls.

During this reporting period, 73 one-pound cans of the reference powder, packaged in high-purity nitrogen, and 8 one-pound cans of the same powder, packaged in vacuum, have been received from Climax. Separately, Climax has also forwarded 2 cans of the nitrogen-packed powder and 2 cans of the vacuum-packed powder to the Aeronautical Materials Laboratory, Naval Air Engineering Center. Chemical analyses of the powder are being performed by Climax and NAEC. It is anticipated that their results will be available shortly.

B. Preparation of Pressurized Compacts

The design features of a modified four-piece split-tapered compacting die were presented and discussed in Progress Report No. 2. At that time, the four-piece tapered sleeve was in the fabrication stage and has since been completed. Although the design specified that the sleeve bore be 1/2 in., deep grooving of the wall during the gun-drilling operation caused the bore to be 0.523 in. after finish-honing. Therefore, it was decided to initiate fabrication of another four-piece tapered sleeve which would conform to the design specifications. At the time of this report, the sleeve is in the heat-treating stage and should be ready for use shortly.

Meanwhile, the four-piece tapered sleeve having the oversize bore (0.523 in.) and a mating plunger were both completed and used to form

MoS₂ compacts. Several compacts were made at 150,000 psi with both Type Z and reference MoS₂ powder. There was some warpage associated with these compacts after they were removed from the compacting die, and severe cracks developed in two of the five compacts formed from reference powder. A compact was then formed at 200,000 psi with the reference powder. It also warped a noticeable amount after removing from the die. Since the excessive clearance between the oversize bore in the four-piece tapered sleeve (0.523 in.) and the raised center portion in the base plate (0.500 in.) was a likely cause of nonuniform compacting of the MoS₂ powder, it was decided to insert a hardened steel pin, 3/8-in. length × 0.523-in. diameter, in the lower portion of the bore and pressurize the compact above same. A compact formed in this manner at 200,000 psi appeared to have very good mechanical integrity and had negligible warpage even though its overall length was slightly greater than 2-1/2 in. It was noted that this compact had a very thin "hairline" surface blemish extending along a portion of its length, and since no flaw was observed in the bore of the die, it was assumed that a scratch in the film of MoS₂ adhering to the die wall had been responsible. Therefore, another compact of reference powder formed at 200,000 psi, using the hardened pin in the lower portion of the bore, was made. Again, there was very little warpage of the compact after removal from the die, but the surface blemish was much larger and extended along a considerable length of the compact. Inspection of the die at this time revealed a crack that had opened up in one section of the four-piece tapered sleeve. The crack extended completely through the section of the sleeve but had not propagated to the edge; therefore, the section remained intact. Due to flow of powder into the crack during the pressure cycle, the cracked section was deformed, thus the four-piece tapered sleeve would not fit together properly. Therefore, material was removed from one of the mating surfaces on the cracked section with a surface grinder in order to permit proper assembly. A hardened steel pin, 2-7/8-in. length × 0.523-in. diameter, was machined and can be employed to raise the compact above the cracked area in the die during the pressurization cycle. On the other hand, compacts formed in the upper portion of the four-piece tapered sleeve in this manner are limited in length to approximately 1-1/4 in. Also, the plunger does not receive nearly the support when used as such, and can be easily bent or broken. The upper portion of the four-piece tapered sleeve is not supported by the outer tapered sleeve and has a tendency to separate slightly during pressurization. Because of these inadequacies, only a limited number of compacts were formed in the four-piece tapered sleeve after the crack developed. It should be noted that none of these compacts, nor the two longer compacts formed when using a hardened steel pin in the lower portion of the bore, either warped appreciably or cracked; thus indicating that it is feasible to produce compacts that are essentially straight and exhibit good mechanical integrity with this type of compacting die.

The several MoS₂ compacts that were made served as specimens for obtaining additional preliminary compression test data, compact specific gravity data, and compact hardness data. The results of these tests will be discussed later in the report.

Even though fabrication of a new four-piece split-tapered sleeve having a 1/2-in. bore had been initiated prior to development of the crack in the sleeve having the 0.523-in. bore, it was decided to delay temporarily further work until a reasonable cause of die failure could be found.

After carefully studying the situations involved, it was decided that failure must have been caused by one of the following:

- (1) Poor design which allowed stresses to exceed the ultimate strength of the material and consequently failure resulted.
- (2) A subsurface inclusion or flaw in the material created a stress concentration whereby a crack initiated and propagated to failure.
- (3) Improper heat-treating of the air hardening steel, thus causing a condition whereby the material had subnormal strength.

Further study of the design led to the conclusion that since the four-piece tapered sleeve is contained by the outer tapered sleeve, the only major stresses of consequence are compressive; the compressive strength of the material should be in excess of 200,000 psi. All four of the pieces were subjected to ultrasonic inspection and no subsurface inclusions or laminations, other than the crack, could be detected. Although this does not mean that the crack did not originally initiate from such a flaw in the material, it does give evidence that the material was relatively if not entirely sound before fabrication.

The remaining alternative was that the material had been improperly heat treated. In checking the steel producer's handbook, the following heat treatment was recommended:

- (1) Heat furnace to a temperature of 1525-1600°F.
- (2) Adjust the atmosphere so that it is definitely oxidizing. Excess oxygen of 2 to 3 percent is preferred.

- (3) Place the cold tool, without preheating, in the hot furnace and let it heat naturally until it uniformly matches the color of the thermocouple in the furnace.
- (4) Soak for twenty minutes at temperature, and an additional five minutes per inch of thickness.
- (5) Remove from the furnace and cool in a free circulating air.
- (6) For best combination of hardness and toughness, draw steel at about 350°F to obtain hardness of Rockwell C 59/60.

The organization that performed the heat treating of the four-piece tapered sleeve was contacted, and it was determined that the proper heat-time cycles were used, but a reducing atmosphere, not an oxidizing atmosphere, was used during the hardening cycle. Since the supplier of the steel states that an oxidizing atmosphere should definitely be used during hardening, it is probable that the material has a tendency to undergo hydrogen embrittlement when heat-treated in a reducing atmosphere. Hydrogen embrittlement could account for failure of the tapered sleeve; it is interesting to note that the crack, on the outer surface of the sleeve, had the same appearance as a hydrogen embrittlement failure. On the other hand, the continuation of the same crack to the inner surface (bore) of the sleeve exhibited the appearance of a tensile failure.

Although it is not definitely known what caused initiation of the crack in the tapered sleeve, such precautions as ultrasonic checking the material prior to use, and heat-treating the material under the correct atmospheric conditions are being carried out during fabrication of the four-piece tapered sleeve currently being made.

III. TEST EQUIPMENT

A. General

The design of the basic test apparatus as well as the loading and displacement-measuring arrangements were presented and discussed in Progress Reports 1 and 2. Fabrication of the basic test facility, including a stainless steel vacuum chamber, has been completed and the components have been assembled. Presently, the load rings and deformation-measuring LVDT's are in the calibration stage. Operation of the air bearing as well as alignment of the lower specimen support by use of the Wood's alloy have both been checked and appear to be satisfactory. The capabilities of the rig will not actually be known until it has been employed for making the various tests, and at present the major "holdup" in the program is a lack of suitable test specimens.

Meanwhile, additional testing using the "breadboard" apparatus for performing preliminary compression experiments in air has been carried out. Cathetometer measurements in conjunction with these compression tests have given additional experimental data which will be discussed later in the report. Details on the "breadboard" apparatus and the capabilities of the cathetometer were discussed in Progress Report No. 2.

B. Special Equipment

Two transducer amplifier-indicators which will operate in conjunction with the LVDT's used for measuring specimen load (stress) and specimen deformation (strain) will be employed. These instruments have a bridge circuit with an internally generated 2400-cycle excitation voltage which meets the requirements of the LVDT's. Critical circuits of the amplifier are stabilized by a voltage-regulated power supply, allowing operation over a wide range of line voltages with no effect on the results of the measurement. The two transducer amplifier-indicators will be used for driving the two axes on an X-Y recorder, thus plotting a permanent record of specimen stress vs. specimen strain during tensile, compression, and torsion tests.

The X-Y recorder to be employed will receive paper up to 8-1/2 in. X 11 in. size and has a maximum pen speed of 20 in./sec. The input resistance is one megohm at null on all fixed ranges, while the accuracy is better than 0.2% of full scale. The instrument has eleven calibrated voltage ranges which vary from 0.5 mv/in. to 50 v/in.

C. Other Equipment

A Rockwell superficial hardness tester equipped with a 1/8-in. ball indenter and only 4.5 kg load was employed for making hardness measurements on the MoS₂ compacts. It is realized that this small load and large indenter diameter differ vastly from any of the standard hardness testing setups using the superficial hardness tester, but the extremely soft nature of MoS₂ requires such. Also, this combination of load and indenter size should be immaterial since only relative hardness measurements are desired. A number of hardness measurements have been made using this arrangement, and meaningful results have been obtained.

IV. EXPERIMENTAL RESULTS

A. General

The amount of experimental work performed on the MoS₂ specimens has been somewhat limited because of the difficulties in producing a sufficient number of acceptable compacts. As explained previously, fabrication of a suitable compacting die has proven to be a difficult task and has required considerable effort.

During this report period, compacts were made with both reference and Type Z MoS₂ powder. Compacts made from the reference material were used for performing compression tests with the "breadboard" apparatus. The cathetometer was employed in conjunction with the dial indicators for making specimen strain measurements during these tests. Prior to the compression tests, these compacts were also used for determining the specific gravity variation along the specimen length. Machining of the specimens to various length-to-diameter ratios provided the specific gravity variation data.

Several compacts were made with Type Z MoS₂ powder, purchased on the open market, in an effort to determine the compact specific gravity variation when shorter compacts were made. Hardness tests were also performed on these compacts.

It should be noted that the specific gravities calculated for the various compacts or specimens are based on measured dimensions and weights of same, and the presence of any impurities in the MoS₂ have not been taken into account. Presumably, allowing for impurities would increase the calculated specific gravities slightly, depending on the nature and amounts of impurities. Since analytical data on the reference MoS₂ have not been received from either Climax Molybdenum Company or Aeronautical Materials Laboratory, Naval Air Engineering Center, the impurities are unknown and will have to be taken into consideration when these data are received.

B. Experiments on Compacts of Reference MoS₂

Eight compacts in excess of 2-in. length, of reference powder, were formed prior to failure of the tapered die sleeve. After measuring the dimensions and weights of these as well as calculating the specific gravities, several specimens were machined from five of the compacts. These specimens were machined from various portions of the compacts, with the

aid of a lathe, and had length-to-diameter ratios varying from 0.972 to 4.44. Data on both the compacts and specimens machined from these compacts are shown in Table 1. It is interesting to note that all specimens machined from the top portion of compacts had specific gravities greater than the compacts from which they were machined, and similarly all specimens machined from the bottom portion of the compacts had specific gravities less than those for the original compacts. On the other hand, a specimen machined from an entire compact or the center portion of a compact had a specific gravity nearly identical to the specific gravity of the compact from which it was machined. This trend indicates that the specific gravity of a compact varies from a maximum at the end which is in contact with the die plunger to a minimum at the end resting on the die base during the compacting process. Evidently, friction during compacting is of major significance in this phenomenon. Other evidence that tends to verify this happening is that the powder tends to compact in a "dish-shaped" orientation that is concave upwards. In other words, a compact that is broken by applying a bending moment will always have fractured surfaces that are concave toward the end that was in contact with the advancing plunger. The magnitude of this concaveness seems to be lessened by vibrating the die after it has been charged with MoS_2 powder and prior to applying pressure. Also, the compacts appear to warp less when the compacting die is vibrated in this manner.

Six of the specimens tabulated in Table 1 were employed for making compression tests, in room air, using the "breadboard" apparatus discussed in Progress Report No. 2. These specimens had specific gravities varying over a range of 4.12 to 4.30 and length/diameter over a range of 0.972 to 3.23. A hysteresis loop was obtained by increasing the applied force up to somewhat below the fracture point and subsequently returning to a state of zero compression. Observation of the hysteresis behavior in Figures 1 and 2 (dashed curves) shows that the stress-strain curves represent a combination of elastic and anelastic strain. The anelastic strain was not fully recoverable, but in each case the apparatus was adjusted such that the stress-strain curves for the second compression, until fracture, started at the origin. It is interesting to note that strain of the specimens as measured by using the cathetometer agree very well with that measured by using the dial indicators. Therefore, it appears that the cathetometer will be of value in obtaining information on "barreling" and "necking" of specimens during compression and tension tests, respectively. Since the results on the second compression as shown in Figures 1 and 2 differ significantly from these on the initial compression, it appears that a specimen cannot be cycled without getting different results. Also, there is evidence that the slope of the stress-strain curve for a specimen will increase with repeated cycling. This can be checked in more detail as soon as the more sophisticated test apparatus is put into operation. The data for the remainder of the

TABLE 1 MEASURED DATA FOR COMPACTS AND SPECIMENS
MADE FROM REFERENCE MCS₂

Compact No.	Compacting Pressure, psi	Compact Length, in.	Compact Diameter, in.	Compact Weight, gm	Compact Specific Gravity	Specimen No.	Specimen Length, in.	Specimen Diameter, in.	Specimen Weight, gm	Specimen Specific Gravity	Remarks
1-R	150,000	2.383	0.534	36,711	4.20	1-R	2.194	0.494	28,935	4.20	Machined entire compact.
2-R	150,000(a)	--	--	--	--						
3-R	150,000	2.395	0.534	36,760	4.17	3-R-center	1.238	0.452	13,392	4.12	Machined center portion of compact.
4-R	150,000(a)	--	--	--	--						
5-R	150,000	2.378	0.534	36,393	4.16						Compact was presented to project engineer as an example.
6-R	200,000	2.510	0.534	39,388	4.27	6-R-upper 6-R-lower	1.280 0.839	0.463 0.481	15,097 10,450	4.28 4.19	Machined top portion and lower portion of compact separately.
7-R	200,000	2.179	0.535	34,326	4.28	7-R-upper 7-R-lower	0.490 1.524	0.504 0.472	6,901 18,608	4.30 4.25	Machined top portion and lower portion of compact separately.
8-R	200,000	2.312	0.537	36,381	4.24	8-R-upper 8-R-center 8-R-lower	0.546 0.650 0.433	0.496 0.499 0.489	7,406 8,832 5,557	4.29 4.24 4.17	Machined top portion, center portion, and lower portion of compact separately.

(a) Large crack developed in compact after removing from die, therefore, measurements could not be made.

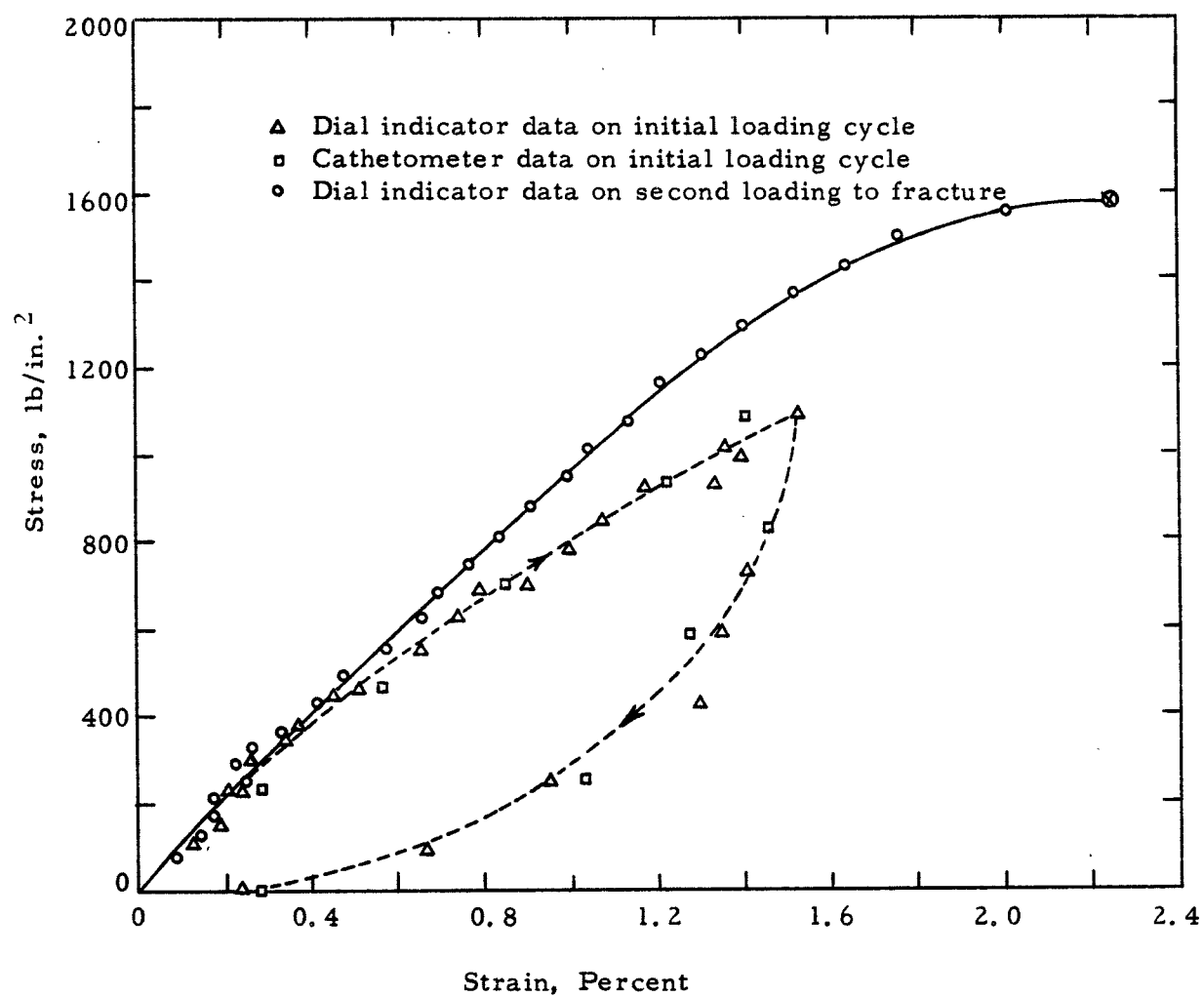


FIGURE 1. HYSTERESIS LOOP RESULTING FROM APPLICATION AND SUBSEQUENT RELEASE OF COMPRESSIVE FORCE ON MoS_2 SPECIMEN

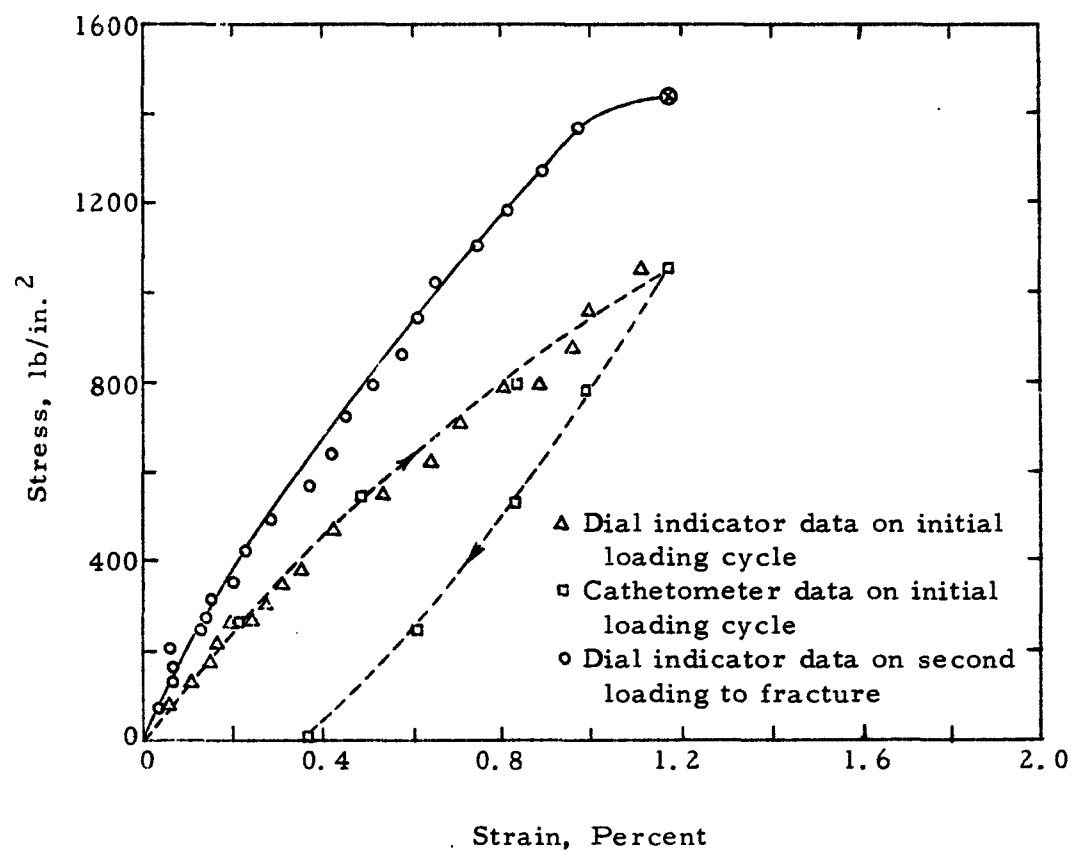


FIGURE 2. HYSTERESIS LOOP RESULTING FROM APPLICATION AND SUBSEQUENT RELEASE OF COMPRESSIVE FORCE ON MoS₂ SPECIMEN

compression tests appear very much as those presented in Progress Report No. 2, and again the slope of the stress-strain curves appear to be a function of specimen specific gravities.

In view of the fact that the data from these tests appear to be similar to those already presented, it was decided to group all of the results and try to arrive at a relationship between specimen fracture stress, specimen specific gravity, and specimen length-to-diameter ratio. It is realized that the specimens employed during this report period were made of reference MoS₂ powder while those used during the previous report period were of Type Z material, and any difference in the analysis of the two could be of major significance. On the other hand, the two types of powder were of the same origination and were probably processed in much the same manner.

Proceeding on the assumption that the data can be considered as representing a single group of tests, the dimensionless parameter $\frac{(S. G. Theo. - S. G. Actual)}{S. G. Theo.}$, where S. G. Theo. is the theoretical

Spec. Length/Spec. Diameter specific gravity of MoS₂ and S. G. Actual is the calculated specific gravity of specimen, was plotted against the fracture stress for each compression test specimen. From this plot it was apparent that the specimen length-to-diameter ratios were not nearly as significant as the specimen specific gravities. Therefore, several deviations of the dimensionless parameter were tried until $\frac{(S. G. Theo. - S. G. Actual)}{S. G. Theo.} (\text{Spec. Length/Spec. Diameter})^{1/8}$ appeared to give a

fairly good fit of the data. This parameter plotted against fracture stress of specimen in compression is shown in Figure 3, and the best straight line has been drawn through the data points by the method of least squares. The 90-percent confidence limits for the ordinate to the regression line are shown by the curved lines plotted in Figure 3. Assuming that this relationship is valid would indicate that the compressive strength of a specimen, having the theoretical specific gravity of MoS₂, would be between 2280 and 3600 lb/in.² 90 percent of the time.

C. Experiments with Compacts of Type Z MoS₂

As discussed earlier in the report, specific gravity of a compact appears to be, at least partially, controlled by the length of the column of MoS₂ being compacted. This must be a function of both the wall friction in the die and the flow characteristics of the compacting media. Therefore, it was decided to run a limited number of experiments, of an exploratory nature, whereby the only varying parameter would be the length of the compact. Six compacts having lengths between 0.307 in. and 1.242 in. were formed from Type Z powder at a pressure of 150,000 psi for 30 minutes.

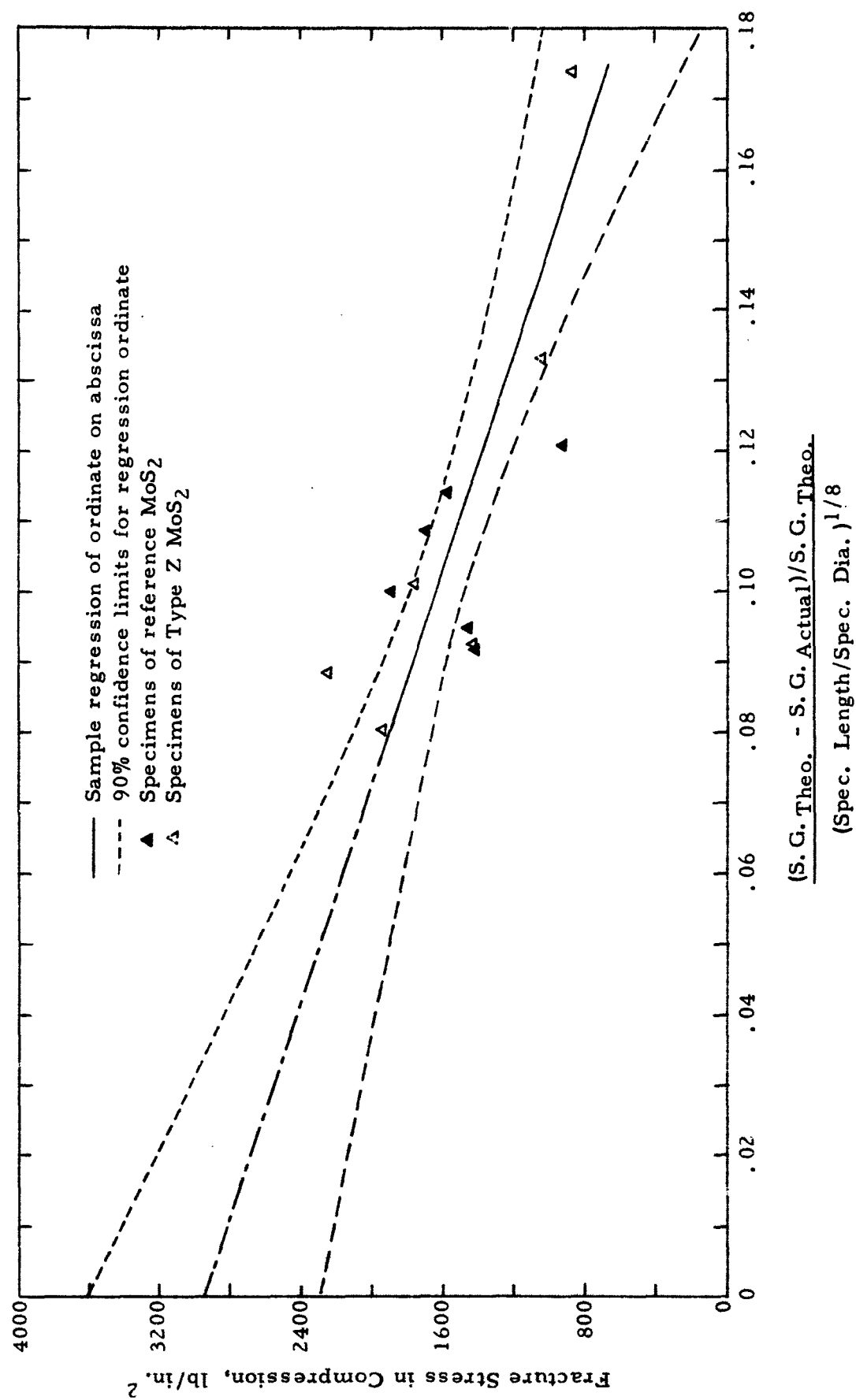


FIGURE 3. COMPRESSIVE STRENGTHS OF MoS₂ SPECIMENS
MADE FROM REFERENCE AND TYPE Z MATERIALS

Four of the compacts that were less than one inch in length had specific gravities of 4.39-4.40 while the longest compact exhibited a specific gravity of 4.36. This evidence together with the data given in Table 1 indicate that the specific gravities of compacts formed in a die similar to the one employed in this work and having length-to-diameter ratios up to unity are essentially constant. On the other hand, there seems to be an inverse relationship between specific gravities and the length-to-diameter ratios for compacts having length-to-diameter ratios substantially greater than unity.

Five hardness measurements, using a Rockwell superficial hardness tester with 1/8-in. ball indenter and 4.5-kg load, were made on each end of these six compacts. There definitely was a difference between the hardness measurements on each end of each compact, the harder measurements being on the end that was in contact with the plunger during compacting. It is interesting to note that the longest compact which had the lowest specific gravity also exhibited the lowest hardnesses. Hardness measurements were not made along the length of the compacts, but it is anticipated that these should provide useful information and will be made in the near future.

V. FUTURE PLANS

Upon completion of the four-piece split-tapered sleeve, pressurized compacts will be made with reference MoS₂ powder, both in "as received" form and after purification to remove water and organic solubles. After compacts have been made, their pertinent properties such as specific gravity and hardness will be measured.

Meanwhile, the test apparatus will be instrumented and calibrated. Then specimens, either machined or not depending on straightness and mechanical integrity of the compacts, will be used in conducting compression, tension, and torsion experiments in clean dry air.

Chemical analysis of the reference MoS₂ powder, both in "as received" form and after purification, will be made and these will be reported along with the results received from Climax Molybdenum Company and Aeronautical Materials Laboratory.

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